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Indian Standard

SPECIFICATION FOR SILVER NITRATE, PHOTOGRAPHIC GRADE

(*Second Revision*)

UDC 661·857·43 : 771·7

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Indian Standard

SPECIFICATION FOR SILVER NITRATE, PHOTOGRAPHIC GRADE

(Second Revision)

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SPECIFICATION FOR SILVER NITRATE, PHOTOGRAPHIC GRADE

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 31 December 1980, after the draft finalized by the Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first issued in 1963 and revised in 1974. In the first revision additional requirements for moisture, reaction with ammonia, nitrites and sulphates were introduced. Taking into account the development made in the country, it has been again revised. In the present revision, additional requirements for copper and reducing substances have been included. Changes have been made in the requirements of moisture, lead and iron content.

0.3 The committee responsible for the preparation of this standard felt that for assessing the quality of silver nitrate required for different types of photographic emulsions, photographic tests were necessary to be performed, the details of which would vary depending upon the type of emulsion to be prepared. It was, therefore, decided to restrict the scope of this standard to silver nitrate required for general photographic emulsions.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for silver nitrate, photographic grade for general photographic emulsions.

---

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be in the form of colourless crystals, free from any foreign matter.

2.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods given in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR SILVER NITRATE, PHOTOGRAPHIC GRADE

SL NO. (1)	CHARACTERISTIC (2)	REQUIREMENT (3)	METHOD OF TEST (REF TO CL NO. IN APPENDIX A) (4)
i)	Silver nitrate content, percent by mass, *Min*	99·8	A-2
ii)	Moisture, percent by mass, *Max*	0·2	A-3
iii)	Clarity of 50 percent solution	Clear and colourless, free from sediment and turbidity	A-4
iv)	Reaction with ammonia	To pass test	A-5
v)	Chlorides	-do-	A-6
vi)	Nitrites	-do-	A-7
vii)	Lead (as Pb), percent by mass, *Max*	0·000 05 (0·5 ppm)	A-8
viii)	Acidity	To pass test	A-9
ix)	Iron (as Fe), percent by mass, *Max*	0·001	A-10
x)	Sulphates (as SO_4), percent by mass, *Max*	0·005	A-11
xi)	Copper (as Cu), percent by mass, *Max*	0·000 2	A-12
xii)	Reducing substances (as S), percent by mass, *Max*	0·000 1	A-13

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in suitable dark-coloured containers as agreed to between the purchaser and the supplier.

3.2 Marking — The containers shall be securely closed and legibly and indelibly marked with the following information:

a) Name and mass of the material in the container;
b) Name of the manufacturer and/or his recognized trade-mark, if any;
c) Date of manufacture; and
d) Lot number to enable the batch of manufacture to be traced from records.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of Indian Standards Institution (Certification Mark) Act and the rules and regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturer or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2)

METHODS OF TEST FOR SILVER NITRATE, PHOTOGRAPHIC GRADE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

---

*Specification for water for general laboratory use (*second revision*).

A-2. DETERMINATION OF SILVER NITRATE

A-2.0 General — Two methods are prescribed, namely, gravimetric method and potentiometric method. However, in case of dispute gravimetric method shall be the referee method.

A-2.1 Gravimetric Method

A-2.1.1 *Reagent*

A-2.1.1.1 *Dilute hydrochloric acid* — approximately 1N.

A-2.1.2 *Procedure* — Weigh accurately about 1 g of the material and dissolve it in 150 ml of water. Heat the solution to boiling and add slowly, with constant stirring, 10 ml of dilute hydrochloric acid. Boil until the precipitate has coagulated and set aside in the dark to cool. Filter through a weighed Gooch or sintered glass (G No. 4) crucible and wash the precipitate with cold water until free from chlorides. Dry the crucible at about 130°C to constant mass.

A-2.1.3 *Calculation*

$$\text{Silver nitrate (Ag } NO_3 \text{), percent by mass} = 118{\cdot}5\ \frac{M_1}{M_2}$$

where

M_1 = mass in g of the precipitate, and

M_2 = mass in g of the material taken for the test.

A-2.2 Potentiometric Method

A-2.2.1 *Reagents*

A-2.2.1.1 *Dilute nitric acid* — approximately 5 N.

A-2.2.1.2 *Ammonium thiocyanate solution* — 0·1 N

A-2.2.2 *Procedure* — Weigh accurately about 5 g of the material and dissolve in water in 250-ml volumetric flask. Make up to mark with water and mix. Pipette out 25 ml aliquot in a beaker. Add 20 ml of water and 5 ml of dilute nitric acid. Keep on a magnetic stirrer and start stirring. Titrate with ammonium thiocyanate solution potentiometrically using silver and mercury or mercurous sulphate electrodes. Titrate in 0·1 ml increments near the end point.

A-2.2.3 *Calculation*

$$\text{Silver nitrate, percent by mass} = \frac{V \times N \times 169 \cdot 9}{M}$$

where

V = volume in ml of thiocyanate solution consumed,

N = normality of thiocyanate solution, and

M = mass in g of the material taken for the test.

A-3. DETERMINATION OF MOISTURE

A-3.1 Procedure — Weigh accurately about 10 g of the material in a petri dish and dry in an oven at 110°C for three hours. Cool in a desiccator and weigh. Repeat the process of heating and cooling until constant mass is obtained.

A-3.2 Calculation

$$\text{Moisture, percent by mass} = \frac{(M - M_1)}{M} \times 100$$

where

M = mass in g of the material before drying, and

M_1 = mass in g of the material after drying.

A-4. TEST FOR CLARITY OF 50 PERCENT SOLUTION

A-4.1 Procedure — Dissolve 100 g of the material in 200 ml of water in a 250-ml beaker. In another beaker, take 200 ml of water. View the solution from above against black and white background. The material shall be considered to have passed the test if the solution of the material as compared against the blank is clear and colourless, and free from sediment and turbidity.

A-5. TEST FOR REACTION WITH AMMONIA

A-5.1 Procedure — Dissolve 30 g of the material in water to get a final volume of 60 ml. Add 80 ml of concentrated ammonium hydroxide and mix well. Prepare another blank solution with 60 ml of water to which 80 ml of concentrated ammonium hydroxide has been added. Compare the two solutions. The material shall be taken to have passed the test if the opalescence of the solution is not greater than that of the blank.

A-6. TEST FOR CHLORIDES

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — 100 ml capacity.

A-6.2 Reagents

A-6.2.1 *Dilute Nitric Acid* — approximately 4 N.

A-6.2.2 *Silver Nitrate Solution* — approximately 10 percent.

A-6.3 Procedure — Dissolve 10 g of the material in 100 ml of water, add 1 ml of dilute nitric acid and dilute in a Nessler cylinder to the mark. In another Nessler cylinder, take 1 ml of dilute nitric acid and dilute to the mark. Allow to stand for 10 minutes. Observe from the top of the cylinders against black background.

A-6.3.1 The material shall be taken to have passed the test if no opalescence is produced with the material.

A-7. TEST FOR NITRITES

A-7.1 Test Reagent — Dissolve 0·1 g of alpha naphthylamine in 75 ml of water. Add 30 ml of glacial acetic acid. Prepare another solution of 0·5 g of sulphanilic acid in 70 ml of water and 30 ml of glacial acetic acid. Mix the two solutions in equal quantities when required.

A-7.2 Procedure — Dissolve 6 g of the material dried in a vacuum desiccator in 50 ml of water in a glass stoppered cylinder. Add 2 ml of the test reagent and mix well. Allow to stand in the dark for 10 minutes. Similarly prepare a blank solution with 50 ml of water and 2 ml of the reagent.

A-7.2.1 The material shall be taken to have passed the test if the pink or red colour produced with the material is not darker than that produced with the blank solution.

A-8. TEST FOR LEAD

A-8.1 Procedure — Dissolve 10·0 g of the material, accurately weighed, in 100 ml of boiling water and add 25 ml of dilute hydrochloric acid. Allow the mixture to cool and filter in a porcelain dish. Evaporate the filtrate to dryness and dissolve the residue with 5 ml of dilute hydrochloric acid. Determine lead by the method prescribed in IS : 7017-1973*.

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*Method for colorimetric determination of traces of heavy metals by dithizone.

A-9. TEST FOR ACIDITY

A-9.1 Reagents

A-9.1.1 *Sodium Carbonate Solution* — 0·01 N, freshly standardized.

A-9.1.2 *p-Nitrophenol Indicator Solution* — Dissolve 0·1 g of *p*-nitrophenol in 100 ml of water.

A-9.2 Procedure — Dissolve 25 g of the material accurately weighed in 200 ml of water in a conical flask. Add 20 drops of *p*-nitrophenol indicator and titrate against sodium carbonate solution to a yellow colour. Similarly, carry out a blank on 200 ml of water.

A-9.2.1 The material shall be taken to have conformed to the requirements of the test if not more than 7·0 ml of 0·01 N sodium carbonate solution has been consumed in the test.

A-10. TEST FOR IRON

A-10.1 Apparatus

A-10.1.1 *Nessler Cylinders* — 100 ml capacity.

A-10.2 Reagents

A-10.2.1 *Dilute Hydrochloric Acid* — 1 : 1.

A-10.2.2 *Ammonium Thiocyanate Solution*

A-10.2.3 *Iso-amyl Alcohol*

A-10.2.4 *Iso-amyl Acetate*

A-10.2.5 *Potassium Permanganate Solution* — 0·1 N.

A-10.2.6 *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [$Fe(NH_4)_2.(SO_4)_2.6H_2O$] in 10 ml of dilute sulphuric acid (10 percent, v/v) and dilute with water to 1 000 ml. Pipette out 10 ml of the solution and dilute with water to make up the volume to 100 ml. One millilitre of this solution contains 0·01 mg of iron (as Fe).

A-10.3 Procedure — Dissolve 5·0 g of the material in 75 ml of boiling water and add 25 ml of dilute hydrochloric acid. Allow the mixture to cool and filter in a porcelain dish. Evaporate the filtrate to dryness and dissolve the residue with 5 ml of hydrochloric acid transferring the solution to a 100 ml Nessler cylinder. Add 5 ml of ammonium thiocyanate solution, 5 ml of *iso*-amyl acetate and 5 ml of iso-amyl alcohol. Mix the solution thoroughly and dilute to the mark with water. In another Nessler cylinder prepare the standard for comparison by acidifying 5 ml of standard iron solution with 1 ml of dilute hydrochloric acid and adding one drop of 0·1 N potassium permanganate solution. To this add 5 ml of ammonium thiocyanate solution, 5 ml of *iso*-amyl acetate and 5 ml of *iso*-amyl alcohol. Mix thoroughly and dilute to the mark with water.

A-10.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of any red colour produced in the upper layer of the solution with the material is not greater than that produced in the blank.

A-11. DETERMINATION OF SULPHATE

A-11.1 Apparatus

A-11.1.1 *Nessler Cylinder* — 100 ml capacity.

A-11.2 Reagents

A-11.2.1 *Barium Nitrate Solution* — 10 percent.

A-11.2.2 *Dilute Nitric Acid* — 2 N.

A-11.2.3 *Standard Sulphate Solution* — Dissolve 0·148 g of ignited sodium sulphate (Na_2SO_4) in water and dilute to 1 000 ml. Further dilute 10 ml of this solution to 100 ml. One millilitre of this solution contains 0·01 mg of sulphate (as SO_4).

A-11.3 Procedure — Dissolve 1·0 g of the material in 10 ml of water in a Nessler cylinder and add 1 ml of 10 percent barium nitrate solution and 2 ml of dilute nitric acid and dilute to the mark. Carry out a control test in another Nessler cylinder using 5 ml of standard sulphate solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture.

A-11.3.1 The material shall be taken to have passed the test if the turbidity produced with the material is not greater than that produced in the control test.

A-12. DETERMINATION OF COPPER

A-12.1 Reagents

A-12.1.1 *EDTA — Citrate Solution* — Dissolve 20 g of ammonium citrate and 5 g of disodium salt of ethylene diaminetetracetate in water and dilute to 100 ml with water.

A-12.1.2 *Cresol Red Indicator Solution* — 0·01 percent in water.

A-12.1.3 *Ammonium Hydroxide* — 20 percent (*m/m*).

A-12.1.4 *Sodium Diethyldithiocarbamate Solution* — 1 percent in water, filtered.

A-12.1.5 *Standard Copper Solution* — Clean the surface of a copper wire with sand paper. Dissolve 1·000 g of pure copper in dilute nitric acid. Heat the solution nearly to dryness to drive off the acid. Add about 10 ml of water and heat again nearly to dryness. Take up the residue in water, add 25 ml of 1 N dilute sulphuric acid and dilute to 1 000 ml. Pipette out 10 ml of this solution in a 100-ml volumetric flask and dilute to the mark with water. One millilitre of this solution contains 0·01 mg of copper (as Cu).

A-12.2 Procedure — Weigh accurately 10·0 g of the material and dissolve in 100 ml of boiling water. Add 25 ml of dilute hydrochloric acid. Allow the mixture to cool and filter in a porcelain dish. Evaporate the filtrate to dryness and dissolve the residue with 5 ml of hydrochloric acid. Transfer to a 100-ml volumetric flask and make up the volume to the mark with water. Pipette out 50 ml of this solution into a 150-ml separating funnel. In another separating funnel, take 1 ml of standard copper solution and 25 ml of water. Treat both the funnels as in **A-12.2.1**.

A-12.2.1 Add 10 ml of of EDTA citrate solution and 2 drops of cresol red indicator. Add concentrated ammonium hydroxide until the solution is purple red. Cool and add 1 ml of sodium diethyldithiocarbamate solution. Protecting from direct light, add 10 ml of carbon tetrachloride and shake for 3 minutes. Allow the layers to separate.

A-12.2.2 The material shall be considered not to have exceeded the limits prescribed in Table 1 if the colour developed in the lower layer in the sample solution is not greater than in that in the blank.

A-13. DETERMINATION OF REDUCING SUBSTANCE

A-13.1 Reagents

A-13.1.1 *Standard Sulphide Solution* — Weigh accurately 0·775 g of sodium thiosulphate ($Na_2S_2O_3 . 5H_2O$) and dissolve in 500 ml of water in a volumetric flask. Pipette out 25 ml of this solution and dilute to one litre with water in a volumetric flask. One millilitre of this solution is equivalent to 0·005 mg of sulphide (as S).

A-13.1.2 *Ammonium Hydroxide* — 20 percent (*m/m*).

A-13.2 Procedure — Weigh accurately 10·0 g of the material in a 100-ml conical flask. Add 20 ml of water and dissolve. Let this solution be *A*. In another flask take 5·0 g of the material, dissolve in 20 ml of water. Add 1 ml of standard sulphide solution and mix. Let this solution be *B*. Treat both the flasks as in **A-13.2.1**.

A-13.2.1 Add 14 ml of concentrated ammonium hydroxide with thorough mixing. Keep in a water-bath for 10 minutes. Remove and transfer to separate Nessler cylinders.

A-13.2.2 The material shall be considered not to have exceeded the limits prescribed in Table 1 if the colour developed in solution *A* is not greater than in solution *B*.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF SILVER NITRATE, PHOTOGRAPHIC GRADE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the precautions and directions given in **B-1.1** and **B-1.2** shall be observed.

B-1.1 The sampling instrument and the containers for samples shall be such that the material has no action on them. They shall be clean and dry.

B-1.2 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature and as far as possible, the material shall also be protected from light.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In a single consignment of the material, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment of the material is known to consist of different batches of manufacture or of different sizes of containers, then the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose (n) shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

(*Clause* B-2.2)

LOT SIZE *N*	NO. OF CONTAINERS TO BE SELECTED *n*
(1)	(2)
Up to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
111 and above	10

NOTE — In the case of very small lots where the selection of 3 containers may be uneconomical, the method of judging the conformity of the lot to the requirements of the specification shall be as agreed to between the purchaser and the supplier.

B-2.3 The containers shall be selected at random from the lot and in order to ensure randomness of selection, the procedure prescribed in IS : 4905-1968* may be followed. In case it is not available the following procedure is recommended for use.

> 'Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, 3,......, up to *r* and so on, where *r* is the integral part of *N*/*n*. Every *r*th container thus counted shall be withdrawn to give sample for tests.'

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected according to **B-2.3**, a small representative portion of the material shall be drawn with the help of a suitable sampling instruments.

B-3.2 Out of these portions, equal quantities of the material shall be taken and mixed thoroughly to form a composite sample. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

---

*Methods for random sampling.

B-3.4 All the individual samples and the composite sample shall be transferred to separate sample containers. All the containers shall be sealed and labelled with full identification particulars.

B-3.5 The referee test samples consisting of a composite sample and a set of *n* individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any disput between the two.

B-4. NUMBER OF TESTS

B-4.1 Test for the determination of silver nitrate shall be performed on each of the individual samples.

B-4.2 Test for the determination of all other characteristics given in Table 1 shall be performed on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — From the test results for silver nitrate content, the mean X and range R of test results shall be computed (range being defined as the difference between the maximum and minimum values of test results).

B-5.1.1 The lot shall be declared as conforming to the requirement for silver nitrate if the value of the expression ($X - 0{\cdot}6\ R$) as calculated from the test results is equal to or greater than 99·8.

B-5.2 For Composite Sample — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement specified.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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117/418 B Sarvodaya Nagar	KANPUR 208005	8 12 72
Patliputra Industrial Estate	PATNA 800013	6 28 08
Hantex Bldg (2nd Floor), Rly Station Road	TRIVANDRUM 695001	32 27

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